18 EXTRACTIONS

18.1 Objectives

18.1.1 To familiarize the trainee with the sample extraction methodology

18.2 Modes of Instruction

18.2.1 Self-directed study through reading assignments and worksheets

18.2.2 Practical exercises

18.3 Reference

18.3.1 Moffat, A. C., editor. *Clarke's Isolation and Identification of Drugs*. London: The Pharmaceutical Press, 1986.

18.3.2 Clarke, E. G. C., *Isolation and Identification of Drugs*, London: The Pharmaceutical Press, 1972, Vol. 1, 2.

18.3.3 Higuchi, T. et al. "Ion Pair Extraction of Pharmaceutical Amines" *Analytical Chemistry*, Vol. 39, 1967, p. 974.

18.3.4 Watson, D. G. *Pharmaceutical Analysis* New York: Churchill Livingstone, 1999, pp. 17-47.

18.3.5 Virginia Division of Forensic Science Controlled Substances Procedures Manual, Psilocybin and Mescaline Sections.

18.4 Assignments

18.4.1 Completion of required reading assignments (18.3.4, 18.3.5)

18.4.2 Study questions and practical exercises

18.5 Study Questions

18.5.1 What is a matrix?

18.5.2 What is the difference between recrystallization and precipitation?

18.5.3 Define the following with respect to filtration:

- Supernatant
- Filtrate
- Porosity
- Retentivity
- Speed

18.5.4 Describe how a mixed solvent recrystallization is performed. How is a single solvent recrystallization performed?

18.5.5 Why is it necessary to have at least two test tubes in a centrifuge?

18.5.6 Define the following:

- Unsaturated solutions
- Saturated solutions

- Supersaturated solutions
- Reflux
- Azeotrope

18.5.7 What is the difference between evaporation and sublimation?

18.5.8 What problems may be encountered if ether evaporates to dryness?

18.5.9 What is a dry extraction?

18.5.10 What effect does temperature have on a drug extraction?

18.5.11 Describe how a series of several extractions will be more effective than a single extraction (using the same volume of solvent) considering the concept of partition coefficients.

18.5.12 What problems are encountered by using a large volume of solvent during extractions?

18.5.13 How can water be removed from organic solvents?

18.5.14 What is an emulsion? How can they be prevented and what can be done when one occurs?

18.5.15 What does "salting out" mean?

18.5.16 What does pH stand for? pK_a?

18.5.17 Describe how a pH controlled extraction works explaining equilibriums that are set up between two immiscible solvents.

18.5.18 Describe an extraction scheme that would recover most non-volatile compounds from an unknown.

18.5.19 What types of functional groups cause a compound to be acidic? Basic?

18.5.20 What does amphoteric mean?

18.5.21 Tell whether the drugs listed in Appendix B are acidic, basic, or neutral.

18.5.22 How is morphine best extracted from powder form?

18.5.23 How does hydrogen bonding come into play in liquid-liquid extractions?

18.5.24 What is ion-pairing? Diagram how it works using equilibrium considerations.

18.5.25 What types of factors should be considered in selecting solvents to use in extractions.

18.5.26 What separation advantages does chromatography have over extraction procedures? Disadvantages?

18.5.27 Describe how a soxhlet extractor works.

18.5.28 Describe the acetic acid extraction of psilocybin mushrooms emphasizing areas of concern.

18.6 Practical Exercises

18.6.1 Obtain a Fiorinal with codeine capsule from the TC. Diagram a suitable extraction scheme using acid/base extractions. Perform these extractions to isolate each component into an organic solvent. Confirm each

component utilizing either GC/MS or FTIR.

18.6.2 Obtain a sample of mushrooms from the TC. If none are available, use a standard of psilocybin. Perform the acetic acid extraction as outlined in the procedures manual. Confirm the presence of psilocyn.

18.6.3 Obtain a sample of a cocaine mixture from the training coordinator. Perform a dry extraction, and confirm using FTIR.

18.7 Modes of Evaluation

18.7.1 Written examination

♦ End